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(54) Polyamide resin and its production.

(57) A polyamide resin has a relative viscosity of 2.5 to 6 and 40 to 100% of its end groups are substituted by C<sub>6-22</sub> hydrocarbon groups. The resin may be made by polycondensation in the presence of a monoamine and/or a monocarboxylic acid providing such groups or the resin may be made by polycondensation followed by postcondensation with a monoamine and/or a monocarboxylic acid providing such groups.

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## Polyamide Resin and its Production

Polyamide resins are made by polycondensation and, due to their mechanical properties and mouldability, are widely used as engineering plastics. However it would be desirable to improve their bending resistance and tensile strength at low temperatures, and also to improve their melt fluidity and mould releasability.

According to the invention a polyamide resin has a relative viscosity of 2.5 to 6 and 40 to 100% of the end groups of the resin are substituted by C<sub>6-22</sub> hydrocarbon groups.

The polyamide resin may be made by amide polycondensation conducted in the presence of a monoamine having a C<sub>6-22</sub> hydrocarbon group and/or a C<sub>7-23</sub> monocarboxylic acid, whereby 40 to 100% of the end groups are substituted by a C<sub>6-22</sub> hydrocarbon group and the resin has a relative viscosity of 2.5 to 6.

Alternatively the resin may be made by amide polycondensation followed by condensation with a monoamine having a C<sub>6-22</sub> hydrocarbon group and/or a C<sub>7-23</sub> monocarboxylic acid, whereby 40 to 100% of end groups become substituted by C<sub>6-22</sub> hydrocarbon groups and the relative viscosity is 2.5 to 6.

In addition to conducting the polycondensation, or conducting the post condensation, in the presence of the specified monoamine and/or monocarboxylic acid it may also be conducted in the presence of a diamine and/or dicarboxylic acid. Thus in one process the monocarboxylic acid is used in combination with the monoamine and in another the monocarboxylic acid is used in combination with a diamine, that typically can have 2 to 22 carbon atoms, either alone or with the monoamine. In another process a monoamine may be used in combination

with a dicarboxylic acid, that may have from 3 to 24 carbon atoms.

The invention includes the process for producing a polyamide resin having hydrocarbon end groups of 6 to 22 carbon atoms and having a relative viscosity of from 2.5 to 6, the number of the hydrocarbon group being 40 to 100% of total number of end groups of the polyamide, comprising either the step (A) of subjecting the starting materials for producing a polyamide to polycondensation, and subjecting the thus obtained polycondensate to condensation with (1) a monocarboxylic acid of from 7 to 23 carbon atoms and a monoamine of from 6 to 22 carbon atoms and/or a diamine of from 2 to 22 carbon atoms, (2) a monoamine of from 6 to 22 carbon atoms and a dicarboxylic acid of from 3 to 24 carbon atoms or (3) a monocarboxylic acid of from 7 to 23 carbon atoms or a monoamine of 6 to 22 carbon atoms under a pressure of lower than 400 Torr, or the step (B) of subjecting the starting materials for producing a polyamide to polycondensation in the presence of (1) a monocarboxylic acid of from 7 to 23 carbon atoms and a monoamine of from 6 to 22 carbon atoms and/or a diamine of from 2 to 22 carbon atoms, (2) a monoamine of from 6 to 22 carbon atoms and a dicarboxylic acid of from 3 to 24 carbon atoms or (3) a monocarboxylic acid of from 7 to 23 carbon atoms or a monoamine of from 6 to 22 carbon atoms under a pressure of lower than 400 Torr in the final stage of the polycondensation.

Polyamide resins are obtained by polycondensation of compounds providing appropriate carboxylic acid and amine groups. For instance the resins may be obtained by polycondensation (or copolymerisation) of a lactam having not less than three members ring, a polymerisable  $\omega$ -amino acid or a dicarboxylic acid with a diamine.

Suitable lactams are  $\epsilon$ -caprolactam, enantolactam, capryl lactam, lauryl lactam,  $\alpha$ -pyrrolidone and  $\alpha$ -piperidone.

Suitable  $\omega$ -amino acids are 6-aminocaproic acid,  
5 7-aminoheptanoic acid, 9-aminononanoic acid and 11-aminoundecanoic acid.

Suitable dicarboxylic acids are adipic acid, glutaric acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, undecanedioic acid, dodecanedioic acid,  
10 hexadecanedioic acid, hexadecenedioic acid, eicosanedioic acid, eicosanedienedioic acid, diglycolic acid, 2,2,4-trimethyladipic acid, xylylenedicarboxylic acid, 1,4-cyclohexanedicarboxylic acid, terephthalic acid and isophthalic acid.

15 Suitable diamines for reaction with these include hexamethylenediamine, tetramethylenediamine, nonamethylenediamine, undecamethylenediamine, dodecamethylenediamine, 2,2,4- or 2,4,4-trimethylhexamethylenediamine, bis(4,4'-aminocyclohexyl)-methane and  
20 m-xylylenediamine.

Suitable C<sub>6-22</sub> hydrocarbon end groups for use in the present invention include aliphatic hydrocarbon groups such as hexyl, heptyl, octyl, 2-ethylhexyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, tetradecylene,  
25 pentadecyl, hexadecyl, heptadecyl, octadecyl, octadecylene, eicosyl and docosyl, alicyclic hydrocarbon groups, such as cyclohexyl, methylcyclohexyl and cyclohexylmethyl, and aromatic hydrocarbon groups such as phenyl, toluyl, benzyl and  $\beta$ -phenylethyl.

30 These hydrocarbon groups are incorporated into the resin as at least 40% of the end groups of the resin. Any remaining end groups are provided by, for instance, amino groups and carboxyl groups derived from the other starting material from which the polyamid is made.

The hydrocarbon end group content of the polyamide is measured by gas chromatography after hydrolysing the polyamide with hydrochloric acid. The amino end group content of the polyamide is measured by dissolving the present polyamide in phenol and titrating thereof with an aqueous 0.05 N hydrochloric acid solution. The carboxyl end group content of the polyamide is measured by dissolving the polyamide in benzyl alcohol and titrating the thus obtained solution by an aqueous 0.1N sodium hydroxide solution. The total number of end groups is the sum of the respective numbers of the hydrocarbon groups, amino end groups and carboxyl end groups.

In the novel polyamide, the number of the hydrocarbon end groups is 40 to 100% of the total number of the end groups. If the amount is less than 40% it leads to undesirable reduction of the fluidity of the molten polyamide resin and of the bending resistance and the tensile strength of the polyamide. Although it is desirable to have the number of the hydrocarbon groups of the present polyamide in the vicinity of 100% of the total number of the end groups from the view point of the physical properties of the present polyamide, the production of such a polyamide may be difficult and so it is preferable for the amount of the hydrocarbon groups to be in the range of 40 to 95%, most preferably in the range of 50 to 92%.

The relative viscosity of the novel polyamide measured in 98% sulfuric acid at a concentration of 1% by weight and at 25°C by using the method of Japanese Industrial Standard (JIS) K 6810 is in the range of 2.5 to 6, preferably 2.5 to 5.

If the relative viscosity is below 2.5 the resistance to fatigue of the polyamide may be unsatisfactory and if the relative viscosity is higher

than 6 the polyamide may have poor fluidity in molten state.

The novel polyamides may be made, as mentioned above, using certain monocarboxylic acids and/or  
5 monoamines.

As the monocarboxylic acid of 7-23 carbon atoms, there may be used aliphatic monocarboxylic acids such as enanthic acid, caprylic acid, capric acid, pelargonic acid, undecanoic acid, lauric acid, tridecanoic acid,  
10 myristic acid, myristoleinic acid, palmitic acid, stearic acid, oleic acid, linolic acid, arachic acid and behenic acid; alicyclic monocarboxylic acids such as cyclohexanecarboxylic acid and methylcyclohexane-  
carboxylic acid; and aromatic monocarboxylic acids such  
15 as benzoic acid, toluic acid, ethylbenzoic acid and phenylacetic acid. The acid may be introduced in the form of a derivative which can play the same role as the free acid during the reaction. For instance, acid anhydrides, esters and amides may be used.

20 As the monoamine of 6 to 22 carbon atoms, there may be used aliphatic monoamines such as hexylamine, heptylamine, octylamine, 2-ethylhexylamine, nonylamine, decylamine, undecylamine, dodecylamine, tridecylamine, tetradecylamine, pentadecylamine, hexadecylamine,  
25 octadecylamine, octadecyleneamine, eicosylamine and docosylamine; alicyclic monoamines such as cyclohexylamine and methylcyclohexylamine; and aromatic monoamines such as benzylamine and  $\beta$ -phenylethylamine. The amines are generally primary amines.

30 The hydrocarbon end groups preferably have at least 14, often 14-18, carbon atoms. If necessary, diamine of from 2 to 22 carbon atoms, dicarboxylic acid of from 3 to 24 carbon atoms, monocarboxylic acid of not more than 6 carbon atoms (such as acetic acid or propionic acid)  
35 and/or monoamine of not more than 5 carbon atoms (such as

methylamine or ethylamine) may be used together with the above-mentioned monocarboxylic acid of 7-23 carbon atoms and/or monoamine of 6-22 carbon atoms.

As the diamines of from 2 to 22 carbon atoms, there may be used aliphatic diamines such as ethylenediamine, trimethylenediamine, tetramethylenediamine, pentamethylenediamine, hexamethylenediamine, heptamethylenediamine, octamethylenediamine, nonamethylenediamine, decamethylenediamine, undecamethylenediamine, dodecamethylenediamine, tridecamethylenediamine, hexadecamethylenediamine, octadecamethylenediamine and 2,2,4- or 2,4,4-trimethylhexamethylenediamine; alicyclic diamines such as cyclohexanediamine, methylcyclohexanediamine and bis(4,4'-aminocyclohexyl)methane; and aromatic diamines such as xylylenediamine.

As the dicarboxylic acid of from 3 to 24 carbon atoms, there may be used aliphatic dicarboxylic acids such as malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, undecanedioic acid, dodecanedioic acid, tridecanedioic acid, tetradecanedioic acid, hexadecanedioic acid, hexadecenedioic acid, octadecanedioic acid, octadecenedioic acid, eicosanedioic acid, eicosenedioic acid, docosanedioic acid and 2,2,4-trimethyladipic acid; alicyclic decarboxylic acids such as 1,4-cyclohexanedicarboxylic acid; and aromatic decarboxylic acids such as terephthalic acid, isophthalic acid, phthalic acid and xylylenedicarboxylic acid.

The polycondensation reaction may be initiated while using the above-mentioned starting materials and following a usual method (refer to USP 2,241,322, USP 2,241,323 and USP 2,071,250), and the above-mentioned carboxylic acid and amine may be added at the optional stage during the time from the initiation of the reaction



to the time when the reaction under a reduced pressure is initiated. The carboxylic acid and the amine may be added together or separately.

5 The amounts of the carboxylic acid and the amine used in the reaction generally corresponds to 20 to 60  $\mu$ eq, preferably 25 to 55  $\mu$ eq (1-10 meq, preferably 2-8 meq) of carboxylic groups and amino groups per one gram (one mol) of the total amount of the starting materials used for the present polyamide, respectively.

10 If the amount of the carboxylic acid and the amine is too small, it is not possible to produce the polyamide resin exhibiting the effects of the present invention, and if the amount is too large it is not possible to produce the polyamide of a high viscosity, and the  
15 physical properties of the resin become less satisfactory.

The novel polyamide is substituted by the hydrocarbon end groups, and has a relative viscosity higher than 2.5. In order to produce such a resin, it  
20 is necessary to carry out the reaction in the final stage of the reaction under a pressure of not higher than 400 Torr, preferably, not higher than 300 Torr, more preferably not higher than 200 Torr. If pressure in the final reaction stage is over 400 Torr, it is impossible  
25 to obtain the stated relative viscosity. Pressures lower than 400 Torr are satisfactory. The reaction under the above reduced pressure may be carried out for longer than 0.5 hour, usually from 1 to 2 hours. If postcondensation is being used to introduce the end  
30 groups, this is preferably conducted at the reduced pressure.

The novel polyamide has excellent mechanical properties, particularly in bending resistance and tensile strength at a low temperature, and also excellent  
35 fluidity at a molten state and mould releasability.

Accordingly, the polyamide can be moulded into three-dimensional moulded articles, containers, films, sheets, tubes and filaments suitable for use as the material elements for motorcars, aeroplanes, ships, electric- and electronic apparata and other industrial goods by various well-known methods of moulding such as injection moulding, extrusion moulding, and compression moulding, etc.

Further, by blending the polyamide resin of the present invention with other resins such as polyamide resin other than the present polyamide, polyester resin, polycarbonate resin, polyacetal resin, polyolefin-series resin, polystyrene-series resin and the like, a resin composition having improved physical properties can be obtained.

The following are non-limitative examples and a comparative example.

Examples 1 to 4

After introducing, into a 200-litre autoclave, 60 kg of  $\epsilon$ -caprolactam, 1.2 kg of water and both stearic acid and octadecylamine in the respective amounts shown in Table 1, the autoclave was tightly closed under an atmosphere of gaseous nitrogen, and was heated to 260°C to carry out the reaction for 2 hours under a pressure while stirring the content of the autoclave. Thereafter, the pressure of the autoclave was decreased to the value shown in Table 1, and the reaction was further continued for 2 hours under the reduced pressure.

After introducing gaseous nitrogen into the autoclave to bring the inner pressure of the autoclave to atmospheric, the stirring was stopped, and the reaction product was taken out in a strand state and chipped. The chipped product was treated with boiling water to extract and remove the unreacted monomer therefrom, and the thus treated product was dried.

Test pieces of the product for tensile and bending tests were prepared by moulding in a 3.6-ounce injection-moulding machine (made by TOSHIBA Kikai Co.Ltd., Model: IS-75S) the thus obtained chipped product at a resin (chipped product) temperature of 250°C using a metal mould 10 mm in length, 10 mm in width and 2 mm in thickness and having a temperature of 80°C. The injection time was 5 sec, the cooling time 10 sec, and the total cycle time 18 sec.

10 The fluidity of the resin when molten state was represented by the injection pressure at the time of injection.

The tensile test was carried out by stretching 20 test pieces at a temperature of -10°C under the instant tensile stress of 5 kgW and counting the number of broken test pieces in the 20 test pieces thus treated.

The bending test was carried out by bending 20 test pieces to an angle of 180° at a temperature of -10°C and counting the number of broken pieces in the 20 test pieces thus treated.

20 The mould releasability of the present polyamide was determined by the following procedures.

Continuous injection moulding of the present polyamide was carried out under a chipped resin temperature of 250°C, a metal mould temperature of 80°C, an injection time of 5 sec, a cooling time of 12 sec and a total cycle time of 20 sec while using the metal mould which can form a lattice having the ribs of 5 mm x 4 mm at an interval of 15 mm in a frame of 80 mm square. The number of shots for injection was counted until the release of the moulded polyamide resin from the mould began to deteriorate. Mould releasability was classified by the following criteria and indices:

	Index	Criterion
	A	Continuous injection could be carried out more than 25 shots.
5	B	Mould release began to deteriorate between the 10th shot and the 25th shot.
	C	Mould release began to deteriorate before the 10th shot.

10 The thus obtained results of physical and chemical properties of the test pieces in Examples 1 to 4 are shown in Table 1.

Comparative Example

15 For comparison, a polyamide was produced in the same manner as in Example 1 except for not using both stearic acid and octadecylamine and using acetic acid in an amount corresponding to 10 micro-equivalent of carboxyl group per one gram of the starting materials of the polyamide. The test results are shown also in Table 1.

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Table 1

Item	Example				Comparative Example
	1	2	3	4	
Amount of stearic acid used ( $\mu\text{eq.}/\text{g}$ ) ( $\text{meq.}/\text{mol}$ )	30 3.39	24 2.71	50 5.65	37 4.18	10 (acetic acid used) 1.13
Amount of octadecylamine used ( $\mu\text{eq.}/\text{g}$ ) ( $\text{meq.}/\text{mol}$ )	30 3.39	24 2.71	50 5.65	37 4.18	0
Polycondensation pressure at final stage (Torr)	200	200	10	30	500
Relative viscosity of polyamide obtained ( $\eta_{\text{rel}}$ )	2.86	3.10	2.60	3.03	2.90
Amount of residual end $\text{NH}_2$ group ( $\mu\text{eq.}/\text{g}$ )	22	19	4	7	37
Amount of residual end $\text{COOH}$ group ( $\mu\text{eq.}/\text{g}$ )	21	19	5	8	47
Ratio of blocking functional end groups (%) *1	58	56	92	83	0
Injection pressure ( $\text{kg}/\text{cm}^2$ )	400	500	280	450	500
Mould releasability	A	A	A	A	C
Tensile strength ( $\text{N}^2/20$ )	2	2	1	1	14
Bending resistance ( $\text{N}^2/20$ )	0	0	1	1	13

Notes: \*1 Ratio of the number of introduced hydrocarbon groups to the total number of the end groups of the product. \*2 N means the number of broken specimens by the test.

Example 5

After introducing 60 kg of  $\epsilon$ -caprolactam, 1.2 kg of water, 1.024 g (0.68 mol%) of stearic acid and 209 g (0.34 mol%) of hexamethylenediamine into an autoclave of a capacity of 200 litres, the content of the autoclave was treated by the same procedures as in Example 1 to produce a polyamide. The results are shown in Table 2.

Example 6

After introducing 60 kg of  $\epsilon$ -caprolactam, 1.2 kg of water, 775 g (0.54 mol%) of octadecylamine and 170 g (0.27 mol%) of adipic acid into an autoclave of a capacity of 200 litres, the content of the autoclave was treated by the same procedures as in Example 1 to produce a polyamide. The results are also shown in Table 2.

The thus obtained two polyamides in Examples 5 and 6, respectively showed the same excellent properties concerning mould releasability, fluidity in the molten state and resistance to fatigue as those of the polyamides obtained in Examples 1 to 4.

The results of testing those in Example 6 were also shown in Table 2.

Example 7

In the same manner as in Example 1 except for using 830 g (0.54 mol%) of stearic acid and the polycondensation pressure at the final stage of 50 Torr instead of both stearic acid and octadecylamine and the pressure of 200 Torr, the present polyamide was produced. The results are shown in Table 2.

Example 8

In the same manner as in Example 1 except for using 800 g (0.54 mol%) of octadecylamine and the polycondensation pressure at the final stage of 50 Torr instead of both stearic acid and octadecylamine and the pressure of 200 Torr, the present polyamide was produced. The results are shown in Table 2.

Table 2

Item	Example 5	Example 6	Example 7	Example 8
Amount of carboxylic acid. ( $\mu\text{eq.}/\text{g}$ ) ( $\text{meq.}/\text{mol}$ )	stearic acid 60 6.78	adipic acid 48 5.42	stearic acid 48 5.42	
Amount of amine ( $\mu\text{eq.}/\text{g}$ ) ( $\text{meq.}/\text{mol}$ )	hexamethylenediamine 60 6.78	octadecylamine 48 5.42		octadecylamine 48 5.42
Polycondensation pressure at final stage (Torr)	200	200	50	50
Relative viscosity of polyamide obtained ( $\eta_{\text{rel}}$ )	2.90	3.10	2.80	2.81
Amount of residual end $\text{NH}_2$ group ( $\mu\text{eq.}/\text{g}$ )	22	20	3	51
Amount of residual end $\text{COOH}$ group ( $\mu\text{eq.}/\text{g}$ )	22	21	50	4
Ratio of blocking functional end groups (%) *1	58	54	48	47
Injection pressure ( $\text{kg}/\text{cm}^2$ )	420	500	380	380
Mould releasability	A	A	A	A
Tensile strength ( $\text{N}^{*2}/20$ )	2	2	3	4
Bending resistance ( $\text{N}^{*2}/20$ )	0	0	1	1

Note: \*1 and \*2 are the same as defined in Table 1.

CLAIMS

1. A polyamide resin characterised in that it has a relative viscosity of 2.5 to 6 and 40 to 100% of the end groups of the resin are substituted by C<sub>6-22</sub> hydrocarbon groups.
2. A resin according to claim 1 in which 50 to 92% of the end groups are the said hydrocarbon groups.
3. A resin according to claim 1 or claim 2 having a relative viscosity of 2.5 to 5.
4. A resin according to any preceding claim in which the said hydrocarbon groups have 40 to 80 carbon atoms.
5. A method of making a polyamide resin by amide polycondensation characterised in that the polycondensation is conducted in the presence of a monoamine having a C<sub>6-22</sub> hydrocarbon group and/or a C<sub>7-23</sub> monocarboxylic acid and 40 to 100% of the end groups are substituted by C<sub>6-22</sub> hydrocarbon groups and the relative viscosity of the resin is from 2.5 to 6.
6. A method of making a polyamide resin by amide polycondensation characterised in that the resin obtained by polycondensation is condensed with a monoamine having C<sub>6-22</sub> hydrocarbon groups and/or a C<sub>7-23</sub> monocarboxylic acid and 40 to 100% of the end groups become substituted by C<sub>6-22</sub> hydrocarbon groups and the resin has a relative viscosity of from 2.5 to 6.
7. A method according to claim 5 or claim 6 in which the final stage of the polycondensation is effected at a pressure below 400 Torr, preferably below 300 Torr.
8. A method according to any of claims 5 to 7 in which the amount of monoamine and/or monocarboxylic acid introduced for providing the said hydrocarbon end groups is from 20 to 60  $\mu$ eq per gram of the total amount of the starting materials for producing the polyamide.
9. A process for producing a polyamide resin having hydrocarbon groups of 6 to 22 carbon atoms as end groups



- of said polyamide and showing a relative viscosity of from 2.5 to 6, the number of said hydrocarbon group being 40 to 100% of total number of end groups of said polyamide, comprising the step of subjecting the starting materials for producing a polyamide to polycondensation, and subjecting the thus obtained polycondensate to condensation with (1) a monocarboxylic acid of from 7 to 23 carbon atoms and a monoamine of from 6 to 22 carbon atoms and/or diamine of from 2 to 22 carbon atoms, (2) a monoamine of from 6 to 22 carbon atoms and a dicarboxylic acid of from 3 to 24 carbon atoms or (3) a monocarboxylic acid of from 7 to 23 carbon atoms or a monoamine of from 6 to 22 carbon atoms under a pressure of lower than 400 Torr.
10. A process for producing a polyamide resin having hydrocarbon groups of 6 to 22 carbon atoms as end groups of said polyamide and showing a relative viscosity of from 2.5 to 6, the number of said hydrocarbon group being 40 to 100% of total number of end groups of said polyamide, comprising the step of subjecting the starting materials for producing a polyamide to polycondensation in the presence of (1) a monocarboxylic acid of from 7 to 23 carbon atoms and a monoamine of from 6 to 22 carbon atoms and/or diamine of from 2 to 22 carbon atoms, (2) a monoamine of from 6 to 22 carbon atoms and a dicarboxylic acid of from 3 to 24 carbon atoms or (3) a monocarboxylic acid of from 7 to 23 carbon atoms or a monoamine of from 6 to 22 carbon atoms under a pressure of lower than 400 Torr in the final stage of the polycondensation.

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